# Amperometric Ozone Micro-sensor for external interfacing to own probe systems

## - Operating Instruction 100 m/100 dbar version -

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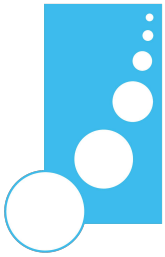

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# Contents

# 1 Preface

This amperometric ES-$O_3$ micro-sensor has been developed above all for the very fast *in-situ* and *online* measurement of ozone and for watching ozone concentrations in industrial applications. For measuring the ozone concentration the sensor has to be combined with a temperature measurement, which is not included in the delivery. Interfacing cables (IL4F) are available in several lengths on customers request.

The working principle of the sensor could be explained simlpy as follows: Because of the partial pressure of the gaseous ozone, the analyte is separated by permeation through the membrane. Inside the sensor the ozone reacts electrochemically at the working electrode. This causes a current corresponding to the partial pressure of the dissolved ozone.

Streaming of the membrane - as it is well-known from all the other membrane covered electrochemical sensors - is not necessary. Due to the micro-sensor technology measurements with high local resolutions of some micrometers are possible. Both turbid and coloured solutions do not interfere with the signal.

The ozone micro-sensor can be used in water depths of up to 100 m resp. 100 dbar pressure. The sensor includes an integrated electronic device for the transformation of the sensor current into a voltage of 0...+ 3 V (DC). The main working range is mostly between 0 and 1 V DC output. The required stabilized power supply is 9...30 V DC.

All sensors are delivered with sensor slope, temperature compensation data and mathematical formulas for calculating the ozone concentration. The exchange of sensor tips is very easy and could be done by the customer himself. The alternative measurement of dissolved oxygen with a separate DO sensor tip extends the sensors flexibility.

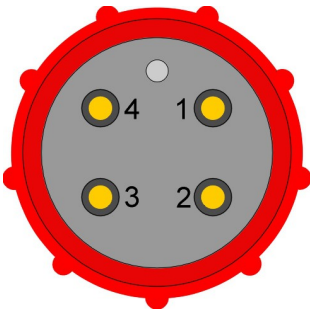
**The perfect functioning and operational safety of the sensor can only be ensured if the user observes the safety precautions as well as the specific safety guidelines stated in the present operating instruction.**

## 2 Technical Data*)

| | |
|---|---|
| sensor type: | amperometric membrane covered micro-sensor |
| power supply: | 9 ... 30 V DC (not included) |
| polarisation time: | approx. 10-20 minutes after switching on, less in case of only short brakes, more in case of natural aged sensors |
| streaming of the membrane: | not necessary |
| stirring of the analyte: | not necessary |
| output: | 0 ... + 3 V DC (main working range approximately 0...1 V DC) |
| connector: | Subconn BH-4-MP (= standard, others on request) |
| materials: | titanium (housing), silicone (membrane), glass (sensor), epoxy resin |
| dimensions: | total length (incl. connector): 234 mm<br>diameter housing with electronic device: 20 mm<br>outer diameter nut: 24 mm<br>outer diameter sensor tip: 13 mm (without o-ring) |
| concentration range: | 0,02....10 mg/l ozone (standard, others on request) |
| accuracy: | ± 2% (measuring value) |
| pressure range: | up to 100 dbar |
| temperature range: | 0°C ... 30°C (for measurement and storage) |
| life time: | approx. 6-10 months depending on the application |
| signal interferences: | $H_2S$<br>signal interferences in case of $H_2O_2$ if the concentration is higher than 2 Vol.% |
| special features: | exchangeable sensor tip (dissolved oxygen), integrated electronic device for transformation of pA-currents into 0 ... + 3 V DC |

*)Changes for technical improvement are reserved.

## Sensor's pin assignment (=sensor's plug view):




Pin 1: ground

Pin 2: not connected

Pin 3: $O_3$-signal output: 0 ... + 3 V DC
(main working range: 0...1000 mV)

Pin 4: power supply 9 ... +30 V DC

# 3 Structure of the ozone micro-sensor

Subconn Connector

Titanium Housing with Electronics

Screw

Sensor Head with Titanium Housing
(=replacement part)

engraved sensor number

Sensitive Part for Measuring

# 4 Putting into operation

For putting into operation please act as follows:

1 Check your plug connections at your probe system/pig tail and compare with the plug connections of the sensor as described in chapter 2.
2 Moisten the sensor plug with some silicone grease and link it with your probe system or pig tail adapter.
3 Switch on the power supply and dive in the sensor into ozone-free water (e.g. tap water or distilled water)
4 Please follow the polarisation for approximately 10-20 minutes and write down the “zero-voltage”, called $U_G$. The sensor is now ready for measurements.

**Attention ! Make sure that the glassy tip will not be destroyed mechanically by handling. This is not covered by the warranty.**

# 5 Measurement and calibration

**Measurement**

Measurements are possible in flow through systems with the AMT flow through cell or *in-situ* by immersing the sensor into the sample solution (in-situ measurements):

*1. in-situ measurement*

- Immerse the sensor/sensor tip into the solution and read the "measuring value" (=U)
- Subtract the residual current ($U_G$), which has to be determined in water (not air) before starting with measurements (see chapter 4: Putting into operation)
- Calculate the ozone concentration as follows:

$c(O_3) = a_{20°C} \times (U-U_G) \times E_T$

| | |
|---|---|
| $c(O_3)$ | dissolved ozone concentration (unit depends on unit for $a_{20°C}$) |
| $E_T$ | temperature correction factor (see calibration sheet last page) |
| $a_{20°C}$ | sensor slope at 20°C |
| $U-U_G$ | measuring value minus "zero voltage" |

*2. flow through measurement*

Insert the sensor into the flow through cell (please order extra). If the O-ring disappears in the flow through cell, the seal is sufficient. Connect now the tube with the analyte solution with the tube of the flow through cell. Put the other tube of the flow through cell into a waste bottle. Now start slowly with pumping the solution through the cell. As flow rate we recommend 1-5 ml/min. Others are possible of course. When filling the flow through cell the first time, take care, that no gas bubbles are enclosed in the flow through cell. Therefore turn round the flow through cell for some seconds, so that the solution output is up. Please take not, that because of adsorption/desorption equilibriums at the vessel and tube walls it may take some time for the adjustment of a steady state. A general rule may be, that you have to exchange the volume of the cell 5-10 times until the adjustment. In case of trace amounts it may take some more time.

Calculate now the ozone concentration as follows:

$c(O_3) = a_{20°C} \times (U-U_G) \times E_T$

| | |
|---|---|
| $c(O_3)$ | dissolved ozone concentration (unit depends on unit for $a_{20°C}$) |
| $E_T$ | temperature correction factor (see calibration sheet last page) |
| $a_{20°C}$ | sensor slope at 20°C |
| $U-U_G$ | measuring value minus "zero voltage" |

## Calibration

For accurate measurements an accurate and periodical calibration is required. The frequency of the calibration depends above all on your demands concerning accuracy and on the chemical matrix of the samples. We offer calibration also as service. Apart from this, new sensors are delivered calibrated, provided that a calibrated sensor was ordered.

If you have ordered a calibrated sensor, you will find at the end of this brochure the sensor slope $a_{20°C}$ and the factors for the temperature compensation ($E_T$). There is also a formula which allows to calculate any $E_T$ at any temperature.

*Self calibration – temperature compensation factors/slope*

If your sensor is not calibrated, first you have to determine the sensor slope $a_{20°C}$ before starting with measurements. In addition - if it's not possible for you to calibrate at the measuring temperature, the temperature correction factors ($E_T$) for your special temperature range have to be determined. For that purpose it is recommended to put the sensor into a flow through cell (available from AMT) and pump slowly (0.8...1.2 ml/min) the $O_3$-solution with a constant concentration through the cell. If the flow through cell is immersed completely into cold water or ice water - a slow increase of temperature with time (or by means of very slow heating on a magnetic stirrer with heating) is realised. You can read the measuring value at any temperature for a constant concentration at the display. After this a simple calculation of $E_T$ is possible.

For determining the sensor slope please act as follows:

- insert the sensor into the flow through cell (please order extra)
- connect the tube with the analyte solution with the flow through cell
- put the other tube end of the flow through cell into a waste bottle
- pump the calibration solution through the cell (recommended: 1-3 ml/min)
- read about 5 different current-concentration pairs (within the range 0...800 mV)
  Do not forget to subtract the residual current from the measured current !
- read the value for the temperature of the solution
- calculate the slope at the measuring temperature (mg/l : mV = $a_{Tm}$) after linear regression
- use the correct $E_T$ (= temperature correction factor according to the enclosed table or to the determined $E_T$'s)
- calculate the $a_{20°C}$ - value by means of equation 1:

$$a_{20^\circ C} = \frac{a_{Tm}}{E_T} \qquad (1)$$

$a_{Tm}$ = sensor slope at measuring temperature; $a_{20°C}$ = sensor slope at 20°C.

For a check up of the sensor slope $a_{20°C}$ act as described before.

Calibration as service is also available from the manufacturer within 1-3 days (plus shipment time).

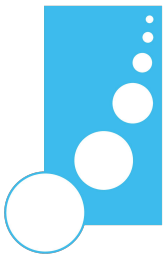
## 6 Maintenance

Mechanical stress of the sensor tip, especially cross forces, unintentional touch downs or strong vibrations have to be avoided. The sensor tip is very weak. Do not touch it. Mechanical damage of the sensor tip excludes that the repair is covered by the guarantee.

For cleaning the sensor tip rinse it in distilled water (if not available, you can take also tap water). Do not use organic solvents like aceton or something like this. Chemical cleaning of the sensor tip is possible by immersing it into diluted HCl (less than 1%) or diluted NaOH (less than 0,02 mol/l) up to maximum of 30 minutes followed by immediate rinsing with distilled water.

Protect the sensor tip with the protection cap during long breaks. Fill the wetting cap with less than ¼ with distilled water to avoid longer adjustment times when starting again the measurements.

## 7 Contact Us



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